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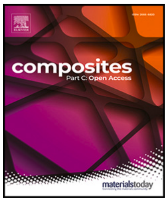

Effect of processing conditions on the tensile properties of PLA/Jute fabric laminates: Experimental and numerical analysis

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ABSTRACT

This article explores how the mechanical properties of composite polymers reinforced with jute fibers are influenced by manufacturing conditions, specifically pressure and temperature. To investigate this, a total of 45 distinct samples were created, and fabricated under nine different pressure and temperature conditions. The results demonstrate a notable linear increase in mechanical properties with incremental changes in pressure, while the impact of temperature variations remains less clearly defined. Based on these findings, a corrective factor was developed for the homogenization formula or rule of mixture that is commonly used to predict the mechanical behavior of composite polymers but does not typically consider manufacturing conditions. The newly introduced corrective factor aims to improve the accuracy of predictions and represents a significant advancement in modeling jute fiber-reinforced composite polymers. This development opens the door for more precise predictions and a better understanding of the intricate relationship between manufacturing conditions and resulting material properties.

1. Introduction

In today's consumer landscape, there is a growing focus on sustainability and environmental consciousness, leading to an increased demand for materials that are eco-friendly, natural, recycled, or biodegradable [1]. This shift has made natural fibers highly appealing as a new category of environmentally friendly materials. These fibers are playing a crucial role in the emerging "green" economy and finding widespread use in contemporary applications [2,3]. Natural fibers are readily available materials found in nature and offer exceptional properties such as biodegradability, cost-effectiveness, high strength, and specific stiffness. Hence, composites reinforced with natural fibers exhibit a range of advantages including reduced weight, cost, toxicity, environmental pollution, and recyclability [4].

Jute, among various natural fiber reinforcing materials, shows promise due to its commercial availability and relatively low cost [5]. Compared to plastics, jute exhibits higher characteristics, making it a viable alternative to conventional fibers in many applications [6]. However, it is important to consider certain characteristics of jute fibers that can influence their mechanical and physical properties. Jute fibers have a multicellular structure composed of microfibrils, and their cross-sections are highly non-uniform that can impact the overall properties of the fibers. Additionally, the mechanical and physical properties of jute fibers can vary significantly. This inconsistency is attributed to factors such as the geographic origin of the jute, the climatic conditions during growth, and the processing techniques used [7]. Other parameters influence as well the mechanical performance of similar fibers, such as the cellulose content, microfibrillar angle, fiber diameter, temperature, presence of defects, and water content within the fibers [8,9].

Furthermore, the processing techniques employed during fiber extraction, along with inconsistent processing methods or inadequate control, can affect the final properties and quality of jute fibers [9]. To address the challenges associated with the inconsistent properties of jute fibers, researchers and industry professionals strive to optimize cultivation techniques, refine processing methods, and control environmental conditions. These efforts aim to enhance the uniformity and reliability of jute fibers for diverse applications, including the automotive, construction, and packaging industries [10–12].

Not only the origin of the natural fibers, its contents and the way of extracting them affect the mechanical properties of the final polymer, but as well the processing technique of producing the composite itself

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by combining the fiber reinforcement and the polymer matrix [13]. On the market, many manufacturing technologies are highly repeatable and capable of achieving process control and delivering high-quality components mainly include extrusion moulding, injection moulding, calendaring moulding, hot pressing moulding, vacuum infusion and others [14].

In relation to the aforementioned factors, it appears that two aspects related to the fibers have an impact on the resulting composite polymer product: firstly, the inherent characteristics of the fibers prior to their application, and secondly, the processing technique employed in creating the composite. However, when studying the mechanical properties of composite polymers, researches have primarily focused on considering only the first factor. Building upon the work of Ishikawa and Chou [15] numerous approaches have been developed for assessing the mechanical properties of two-dimensional 2D and 3D woven composites [16]. These methodologies can be broadly classified into analytical methods grounded in the mechanics of materials approach [17,18], finite element analysis-based techniques [19], and approaches utilizing the asymptotic expansion homogenization method (AEHM) [20,21]. From here, many researchers asked this very essential and fundamental question on how to run and adjust a manufacturing process to achieve a desired product [22]. Some studied the effects on wood plastic composites and showed that injection moulding gives better results than extrusion [23]. Others concluded that the type and parameters of processing techniques have influence on the final properties of the composites [24]. Bernard et al. [25] conducted a study to examine how processing parameters, such as temperature and speed, influence the mechanical properties of plastic composites reinforced with kenaf fiber. Another researchers like Liu et al. [26] showed that compression moulding gives better results than extrusion/injection moulding. Many other researchers conducted similar researches on the effect of the method of manufacturing or on the effect of some parameters [27–29] but a mathematical relations between some of the parameters or the processing technique and the properties of the final product were not faced.

In this study, the researchers focused on examining how the mechanical properties, specifically the Young’s Modulus, of composite materials containing jute fibers and produced by hot compaction, change as a function of compression temperature and pressure cycle. In total 45 different samples, divided into nine groups composed each of five samples, have been tested under three different temperatures and three different pressures. The objective of this research is to discover a mathematical correlation between the used parameters and the mechanical properties of the final product.

2. Manufacturing of the samples

2.1. Materials

The composite polymer used to prepare this paper is composed of a matrix of PLA Luminy LX175 (Polylactic acid resins) and jute fibers. Polylactic acid resins, of renewable origin, allow a significant reduction in the carbon footprint compared to traditional plastics of petrochemical origin [30]. Specifically, PLA Luminy® LX175 has the following characteristics:

- density ρ: 1.24 g/cm^3
- MFI@ 210 °C/2.16 kg: 6 g/10 min
- T_g: 60 °C
- T_m: 155 °C
- Elastic modulus E: 3.5 GPa

Jute fibers supplied by Composites Evolution (https://compositesevolution.com) were used as the fiber reinforcement of the matrix. In particular, it was considered a 290 g/m^2 fabric characterized by density: 1.46 g/cm^3, fiber bundle diameter: 20 μm, elastic modulus E_T: 40 GPa. The plain weave was chosen, see Fig. 1 [31].

2.2. Composite laminate manufacturing

The laminated samples were obtained by film-stacking of plastic films and layers of fibrous reinforcement and hot-pressing of the assembled system [32]. This process, typical of thermoplastic composites, includes three different stages: (1) cutting of fabrics; (2) manual lamination consisting of overlapping plastic films and reinforcement layers in the correct orientation; (3) compaction of the stacked system with the aid of a hot press to ensure adequate impregnation of the fibrous tissue by the molten polymer phase. In this case, the final product is obtained by overlapping 14 layers of woven jute fibers. The compaction was carried out considering three different temperatures: 180 °C, 190 °C and 200 °C and, for each of these, three different pressure cycles were set corresponding to a maximum pressure of 40 bar, 60 bar and 100 bar, respectively.

2.3. Mechanical tests

All the composite samples were characterized experimentally through tensile tests performed on rectangular specimens cut directly from the laminates compacted with the procedure referred to in a previous paragraph 2.2. Tensile tests were performed following ASTM D3039-10 as a guideline [33] using a Universal Testing Machine with the following characteristics:

- Load cell: 100 kN
- Cross-head speed: 2 mm/min
- Grip distance: 100 mm
- Specimen size: 24.4 × 200 mm^2

For every scenario, five samples undergo testing, and the resulting averages are calculated and then used in the rest of the article. The specimen is securely held in the grip, subjected to a load, and the associated deflections are observed. The load is progressively applied until the specimen fractures, at which point the break load and ultimate tensile strengths are recorded. Tensile stress and strain are meticulously documented, and graphs depicting load versus length are generated for analysis.

2.4. The protocol of the test

45 specimens in total have been tested. Every 5 specimens went under the same circumstances of temperature and pressure. These samples have been divided into three different groups: the first group of 15 specimens were tested under a temperature of 180 °C, the second group was manufactured under a temperature of 190 °C, and the last group at 200 °C. Each group was divided into three different sub-groups where the first sub-group was manufactured under a pressure of 40 bar, the second sub-group 60 bar and the third under 100 bar where the pressure was increased 5 bars each 2 min.

2.5. Morphological analysis

The effect of the process conditions on morphological aspects was monitored with photos of the surfaces of the laminates and images collected using a field emission scanning electron microscope (mod. FEI QUANTA 200F) operating in low vacuum conditions.

3. Results

The average results of the tested specimens have been represented in Table 1. Each row represents the average results obtained for 5 different specimens under one temperature and one pressure. Taking as example the results shown in the first row, it means at temperature 180 °C, the maximum pressure applied is 40 bar for all 5 specimens that have an average thickness h of 6.2 mm. The average Young’s Modulus E is 2.53 GPa with a variation of ±0.44. The maximum tensile

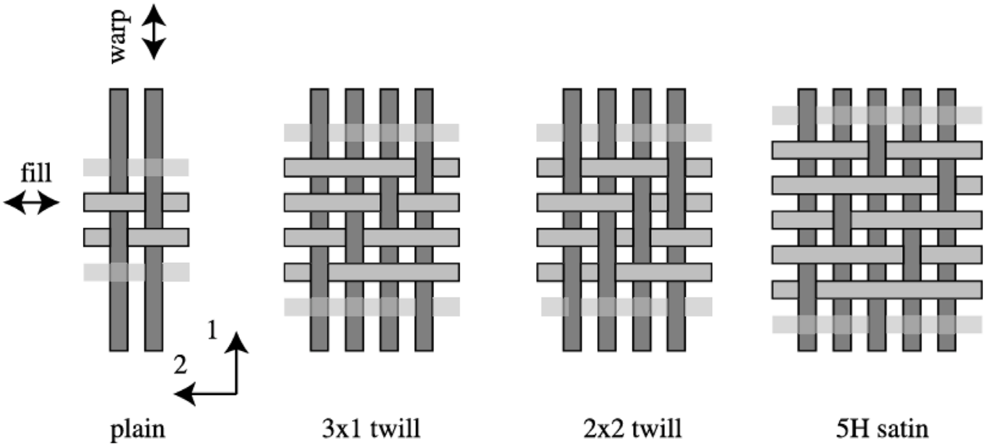

Fig. 1. Schematic representation of four bi-axial woven fabric architectures [31].

Table 1
Results of the experimental campaign of 45 different specimens.

T (°C)	$P_{\max}/h$ (bar)/(mm)	E (GPa)	$\sigma_{\max}$ (MPa)	σ_u (MPa)	$\epsilon_{\max}$ (%)	ϵ_u (%)
180	40/6.2	2.53 ± 0.44	33.75 ± 1.06	16.33 ± 1.10	2.24 ± 0.38	2.30 ± 0.37
	60/6.2	3.76 ± 0.21	36.07 ± 2.49	16.97 ± 1.46	1.81 ± 0.39	2.01 ± 0.24
	100/5.8	6.30 ± 0.50	56.51 ± 6.50	27.52 ± 3.15	1.17 ± 0.26	1.18 ± 0.25
190	40/6.3	2.75 ± 0.08	23.41 ± 0.08	11.05 ± 0.13	1.30 ± 0.09	1.38 ± 0.10
	60/5.8	3.34 ± 0.12	25.05 ± 0.91	12.20 ± 0.36	1.12 ± 0.01	1.81 ± 0.05
	100/5.7	4.55 ± 0.61	24.97 ± 4.51	12.21 ± 2.25	0.72 ± 0.02	0.74 ± 0.03
200	40/6.7	3.07 ± 0.21	23.47 ± 0.78	11.70 ± 0.52	1.04 ± 0.12	1.05 ± 0.12
	60/6.0	3.98 ± 0.23	29.26 ± 0.37	14.71 ± 0.42	0.99 ± 0.08	1.00 ± 0.08
	100/5.0	5.56 ± 0.21	24.73 ± 1.90	12.54 ± 0.92	0.53 ± 0.04	0.54 ± 0.04

stress is 33.75 MPa with a variation of ±1.06. To this maximum stress corresponds an elongation of 2.24% ± 0.38. In what concerns the tensile stress at fracture, the average value is 16.33 MPa ± 1.10, and the correspondent elongation is 2.30% ± 0.37. The mechanical properties of fiber composites can be significantly or slightly influenced by the temperature and pressure conditions during the manufacturing process, hence the study focused on investigating the relationship between the Young's Modulus obtained of the fiber composite under the pressure and temperature conditions during the manufacturing.

3.1. *Effect of pressure*

The results revealed that the Young's Modulus exhibited a linear dependence on the applied pressure, Table 1 and more clearly in Fig. 2. Increasing the manufacturing pressure led to higher values of the modulus. Pressure is essential for consolidating the fiber layers and ensuring proper fiber–matrix bonding. It minimizes void content, enhances fiber wetting, and improves the interfacial strength, thereby positively affecting mechanical properties such as strength and stiffness. Moreover, applying pressure during manufacturing can promote better alignment of the fibers, resulting in improved anisotropic properties of the composite. Controlled resin flow, facilitated by pressure, helps achieve uniform impregnation of the fibers, avoiding regions with excessive or insufficient resin content that could negatively impact mechanical properties. This general declaration is shown by laboratory tests.

At a fabrication temperature of 180 °C, the Young's Modulus was found to be 2.53 GPa when the pressure was 40 bar. As the pressure increased to 60 bar, the modulus increased to 3.76 GPa, and further increased to 6.3 GPa at a pressure of 100 bar. Similarly, at a fabrication temperature of 190 °C, the Young's Modulus values were 2.75 GPa, 3.34 GPa, and 4.55 GPa for pressures of 40 bar, 60 bar, and 100 bar, respectively. When the manufacturing was carried out at a temperature of 200 °C, the relationship between the Young's Modulus and pressure remained linear, with the modulus increasing as the applied pressure increased. The Young's Modulus values were 3.07 GPa, 3.98 GPa, and 5.56 GPa for pressures of 40 bar, 60 bar, and 100 bar, respectively.

Table 2
Results of the Young's Modulus.

Temperature (°C) Pressure (bar)	180	190	200
40	2.53	2.75	3.07
60	3.76	3.34	3.98
100	6.30	4.55	5.56

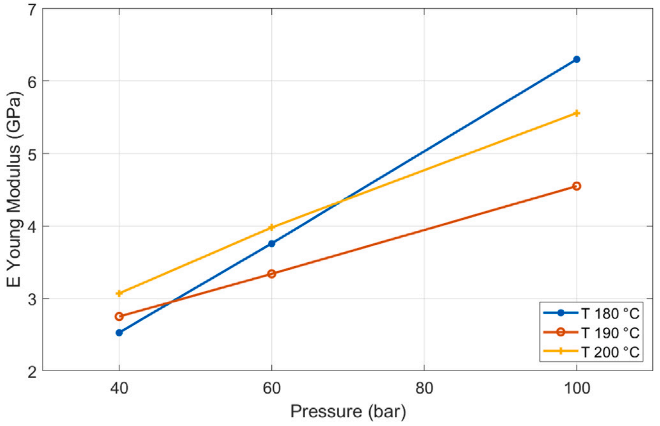

Fig. 2. Relation between Young's Modulus and pressure for fixed temperature.

Referring to what it was mentioned, for an established value of temperature, the values of Young's Modulus increases with the increment

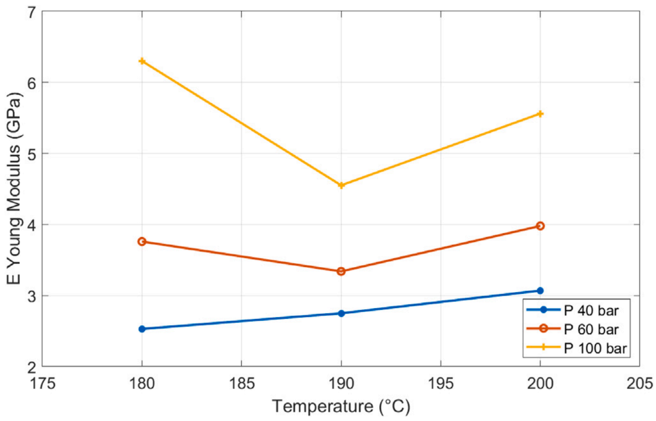

Fig. 3. Relation between Young's Modulus and temperature for fixed pressure.

of the pressure in a linear way. For the values presented in Table 2, each column can be represented by a linear equation slightly different one from the other and shown in Fig. 2, so three different linear equations can be obtained. Using the closed form least-squares regression method, a general increasing linear equation that represent this linear relation, can be written as follow in Eq. (1):

$$E(P) = 0.045P + 1.10 \tag{1}$$

3.2. Effect of temperature

With respect to the temperature effect, in the case of thermoplastic matrices, the temperature mainly influences the viscosity of the melt: the higher the temperature, the greater the fluidity of the melt and, therefore, higher is the quality of impregnation of the fibrous layers by the host matrix. This behavior is reflected in a greater interaction between the phases, more effective transfer of stress and, consequently, can ensure improved mechanical performance of the composite material.

However, too excessive temperatures can also trigger adverse effects in terms of the initiation of thermal degradation processes affecting both the main constituents and the interfaces that can compromise the ultimate performance of the product. Additionally, the differential thermal expansion between the fiber and matrix materials can induce stress within the composite structure. Pressure profiles, on the other hand, have generally a milder effect on the viscosity of the molten matrix. An increase in pressure can favor the densification of the polymer melt, reducing its impregnation capacity but, in a more pronounced way, it can alter the structural integrity of the natural fibers of the reinforcement. It is worth noting that when examining the values from a different perspective, for a fixed pressure, the relationship between the Young's Modulus and temperature was not consistently linear. Based on the laboratory experience, a temperature of 180 degrees yielded the best results for the hot press technique. Higher temperatures tended to reduce the overall quality of the products.

For example, at a pressure of 40 bar, the Young's Modulus values increased with temperature, measuring 2.53 GPa, 2.75 GPa, and 3.07 GPa for temperatures of 180, 190, and 200 °C, respectively. The relationship was linear in this case due to the relatively low pressure. At a pressure of 60 bar, the Young's Modulus values remained relatively stable at 3.76 GPa, 3.34 GPa, and 3.98 GPa for temperatures of 180, 190, and 200 °C, respectively. However, at a higher pressure of 100 bar, the trend of the Young's Modulus values showed a decrease with increasing temperature. For temperatures of 180, 190, and 200 °C, the Young's Modulus values were 6.3 GPa, 4.55 GPa, and 5.56 GPa, respectively. It is important to consider that the specific effects of temperature and pressure on mechanical properties depend on factors such as the composite materials used, the manufacturing process employed, and the characteristics of the fibers and resin system. Regarding the rows of Table 2, it is easy to notice that for a constant pressure, the increment of temperature does not increase necessarily the Young's Modulus. It is possible to notice in Fig. 3, that at 40 bar, the blue representative equation is an increasing linear equation; at 60 bar, the red representative equation can be represented virtually by a constant equation, while the orange equation that corresponds to data at 100 bar can be represented as decreasing linear equation. Using the least square method in order to have one general linear equation that can represent these three different equations in Fig. 3, the result is an almost constant trend line that barely changes with the temperature (2), which indicates the minimum effect of temperature on the Young's Modulus.

$$E(T) = 0.006T + 11.75 \tag{2}$$

3.3. Morphological observations

The analysis of the tensile results was supported by the morphological observations collected both on the surface and on the section damaged by the mechanical test of each laminate sample. For each combination of temperature and maximum compaction pressure, representative images are collected in the following Figs. 4–12. In all cases, at the same temperature, an increment in the level of compaction of the laminate was noted by increasing the pressure. However, at an established compaction pressure, the increment in the temperature was reflected in a slight darkening of the surface: a sign of the triggering of thermo-degradative phenomena affecting both the matrix and the jute fibers. This effect was more pronounced at temperatures 190 and 200 °C, as the pressure increased.

In other words, although a more effective compaction of the composite is expected with the increment of hot-compaction pressure, and by increasing the process temperature beyond (180 °C) that is considered optimal for the reference matrix, the ultimate performance of the composite are determined by a compromise between the level of compaction achieved and the severity of the degradation suffered by the main constituents, matrix and reinforcement.

These considerations allow, among other things, to explain why, for the samples produced by applying the maximum compaction pressure, there is an average decreasing trend of the Young's modulus as the process temperature increases. Hence, the slow increment of the same mechanical parameter with the compaction pressure for process temperatures equal to 190 and 200 °C appears justified compared to the case of laminates produced at 180 °C as it can be noticed from the slope of the equations in Fig. 3.

3.4. Corrective factor for the homogenization equation

It has been observed that the compaction pressure has an increasing linear relation with the Young's Modulus Eq. (1). The effect of temperature is less pronounced as it can be noticed in the slope of Eq. (2) comparing to the slope of Eq. (1). The objective of this study is to find a relation between these two parameters, temperature and pressure of processing, and the Young's Modulus of the fiber composite polymer. Hence, referring always to the least-square method using the data presented in Table 1 and Table 2, and from the interpretation of the previous two initial equations, the following linear equation can be obtained:

$$E(P, T) = 0.063P - 0.037T + 6.66 \tag{3}$$

From this simplified model, the equation demonstrates a linear dependence on pressure, with the impact of temperature being relatively less significant due to the associated coefficient being nearly half that of the pressure coefficient. Nevertheless, it is important to note that this equation is not intended for calculating the Young's Modulus of polymer composites. Instead, its purpose is to serve as a foundation for developing a reduction coefficient. This coefficient can be

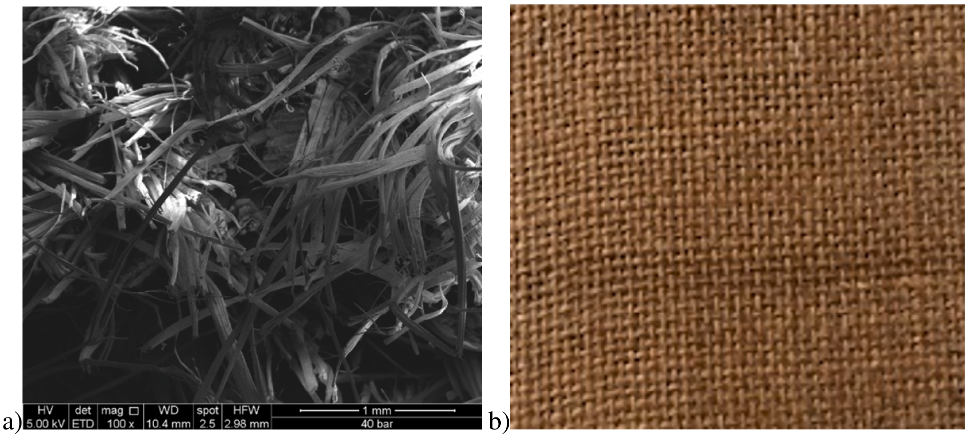

Fig. 4. Close-up view of the surface (a) and SEM micrograph of the tensile damaged section (b) of laminates obtained at 180 °C and 40 bar.

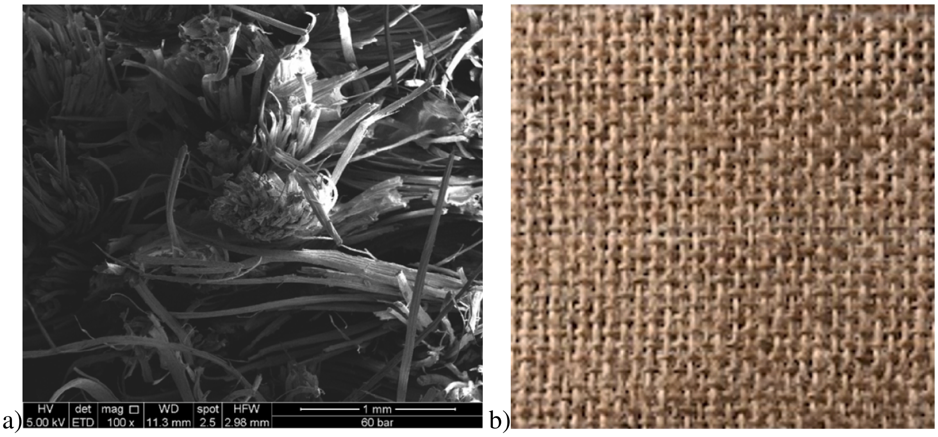

Fig. 5. Close-up view of the surface (a) and SEM micrograph of the tensile damaged section (b) of laminates obtained at 180 °C and 60 bar.

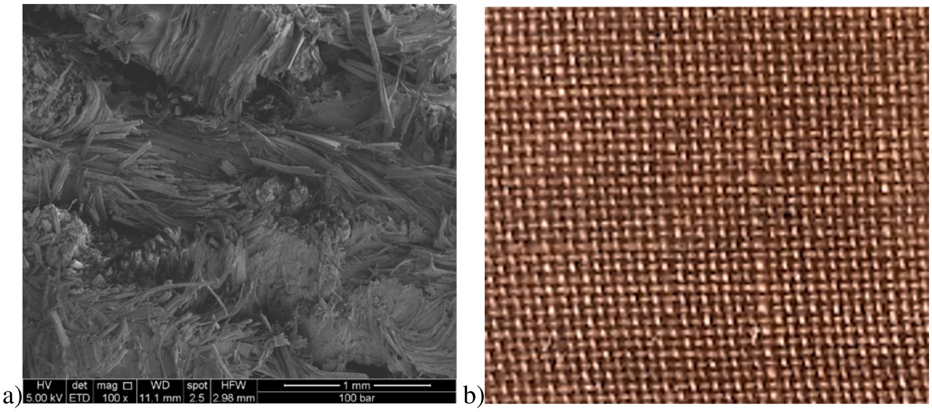

Fig. 6. Close-up view of the surface (a) and SEM micrograph of the tensile damaged section (b) of laminates obtained at 180 °C and 100 bar.

incorporated into homogenization equations to effectively consider the momentary effects of composite polymer manufacturing parameters. The proposal of general corrective factor is still pre-mature because it requires an extensive experimental campaign covering a wider spectre of fibers, natural and synthetic, different fabrication methods, and different number of plies.

The homogenization approach detailed in this article follows the theory of mixture or Chamis approach applied to woven fabrics featuring a fundamental isotropic matrix, as outlined in prior research studies by the authors [34,35]. For the reader's convenience, the equation that depicts the Young's Modulus E of a fiber composite is restated herein Eq. (4). The composition of the composite includes a matrix indicated by the letter m, mainly of plastic, and a fiber, in this case the jute fibers, indicated by the letter f.

$$E = E_1 V_1 + E_2 V_2 \tag{4}$$

where:

$$E_1 = E_f V_f + E_m V_m \tag{5}$$

$$E_2 = E_3 = E_m / (1 - V_f (1 - E_m / E_{2f})) \tag{6}$$

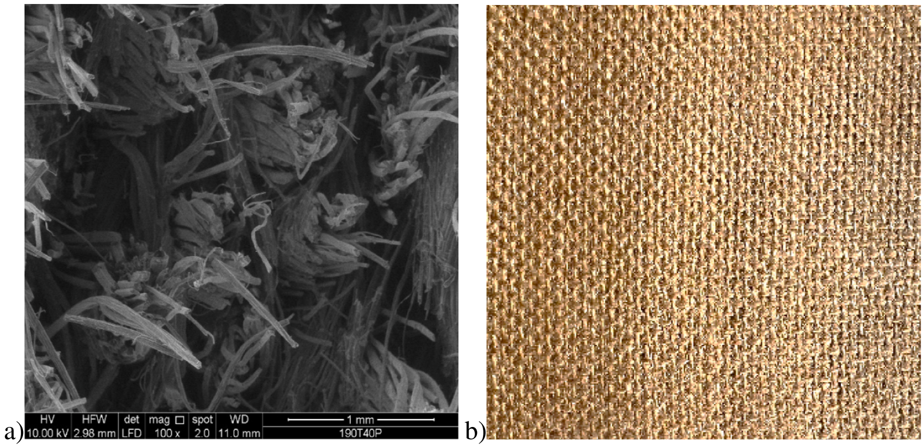

Fig. 7. Close-up view of the surface (a) and SEM micrograph of the tensile damaged section (b) of laminates obtained at 190 °C and 40 bar.

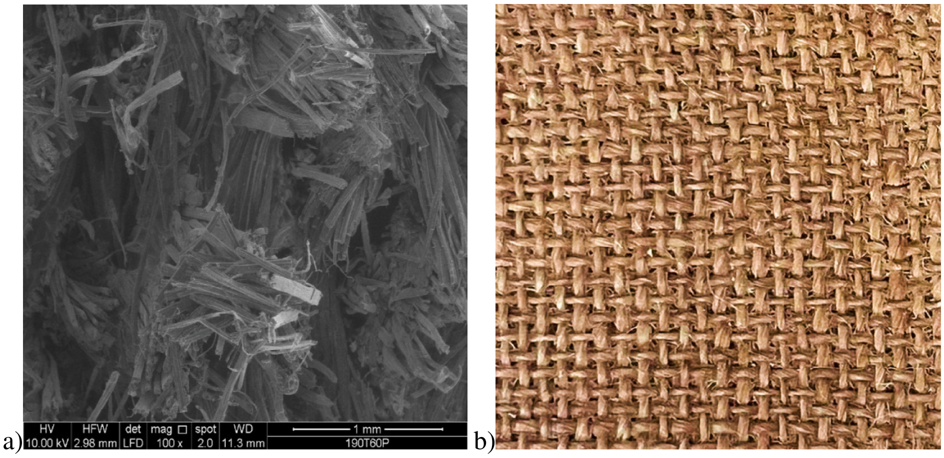

Fig. 8. Close-up view of the surface (a) and SEM micrograph of the tensile damaged section (b) of laminates obtained at 190 °C and 60 bar.

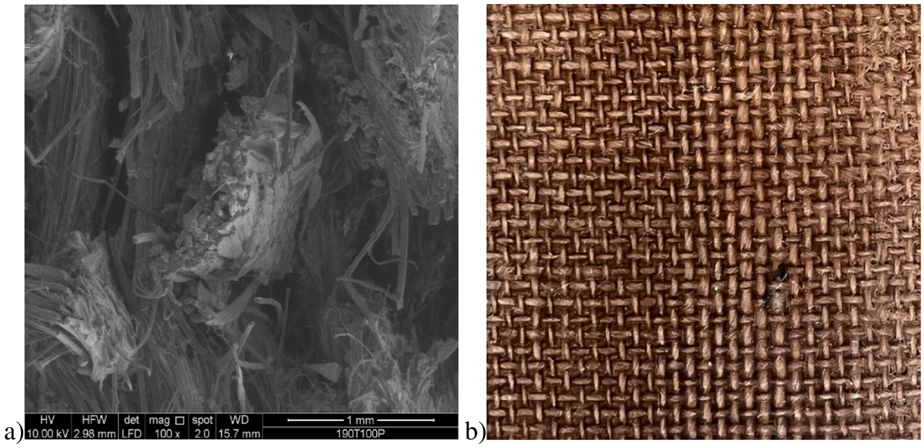

Fig. 9. Close-up view of the surface (a) and SEM micrograph of the tensile damaged section (b) of laminates obtained at 190 °C and 100 bar.

An important step is to consider the volume fraction of each component where $V_f + V_m = 1$, V_f is determined as the volume of the fibers, obtained by the following formula:

$$V_f = \rho_{wf}/(\rho_f h_{wf}) \tag{7}$$

ρ_{wf} represents the nominal areal weight of the Jute Fiber, ρ_f is the density of the fiber and h_{wf} is the thickness of the fabric calculated from the total number of plies of fibers used n multiplied by the thickness of the single fiber.

$$h_{wf} = n\ h_f \tag{8}$$

The used fabric is plain weave or balanced fabric, this means that $E_X = E_Y = (E_1 + E_2)/2$. Based on the preceding findings, it was evident that the Young's Modulus exhibited a distinct variation due to the fibers that has higher Young's Modulus E_f that the PLA matrix that contributes minimally in the total E. This implies that any corrective

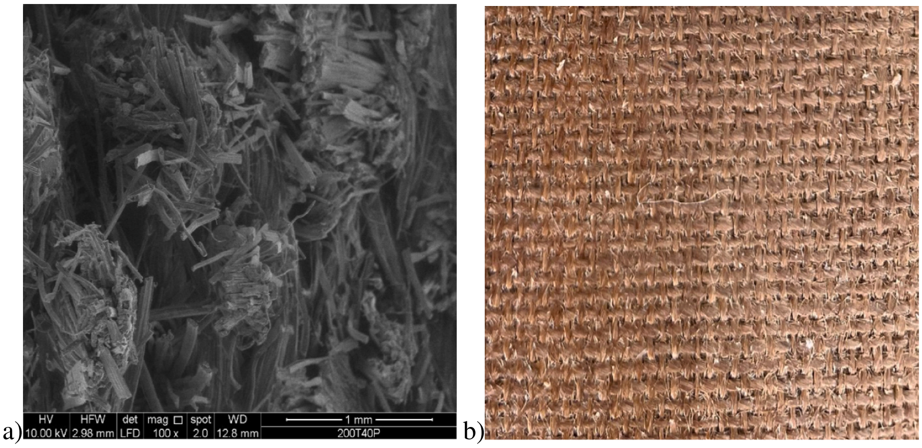

Fig. 10. Close-up view of the surface (a) and SEM micrograph of the tensile damaged section (b) of laminates obtained at 200 °C and 40 bar.

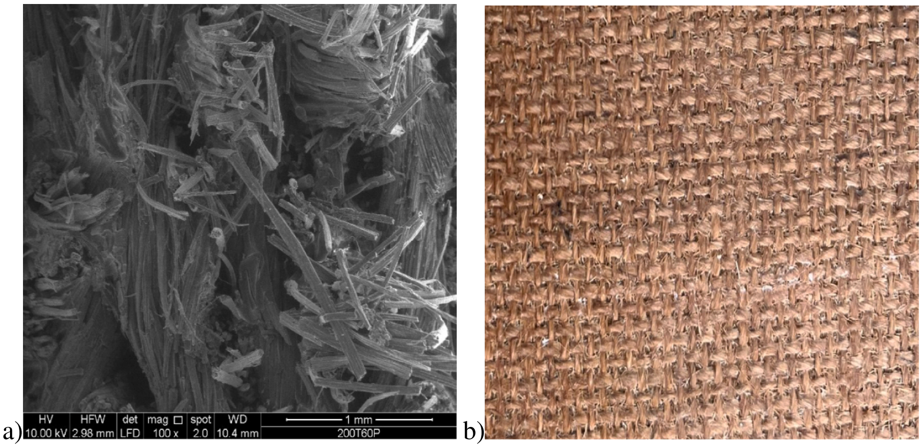

Fig. 11. Close-up view of the surface (a) and SEM micrograph of the tensile damaged section (b) of laminates obtained at 200 °C and 60 bar.

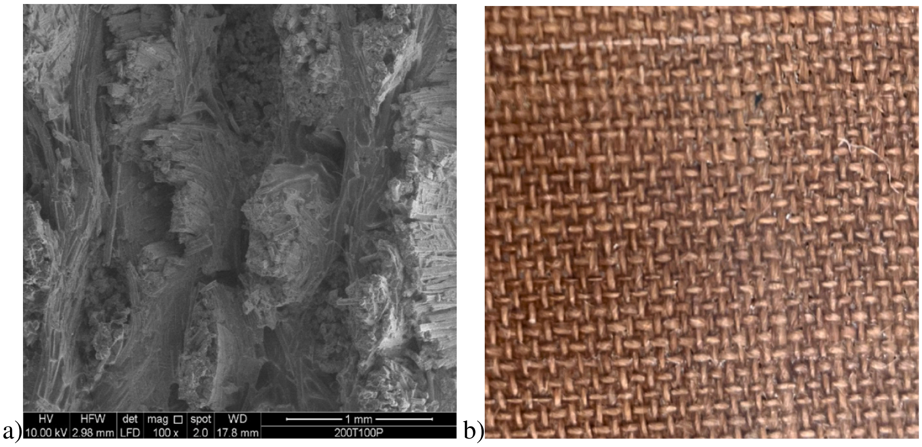

Fig. 12. Close-up view of the surface (a) and SEM micrograph of the tensile damaged section (b) of laminates obtained at 200 °C and 100 bar.

factor introduced should predominantly impact the fiber, particularly its volume of participation, rather than its mechanical properties. From the previous analysis, the new corrective factor will depend on the pressure more than on the temperature. After a linear calculation, the following factor C can be obtained:

$$C = 6.2P - 0.4T + 1.5n \tag{9}$$

where P is the applied pressure, T is the temperature and n is the number of plies. Another factor described as: $C_{Total} = n^2/C$ should multiply V_f in Eq. (7).

This final coefficient represents the equivalent total number of fiber plies that participated effectively in the composite polymer and in increasing its mechanical properties.

4. Conclusion

The static mechanical properties of 45 samples of composite polymers, composed of PLA as matrix and jute fiber as reinforcement, were obtained under various processing (temperature and pressure profile) conditions and systematically characterized in terms of tensile performances.

Key observations include the conspicuous impact of pressure on mechanical properties, showing a linear relationship wherein the properties increase with rising pressure, as indicated by initial calculations. The influence of temperature is less straightforward, with products produced at a lower temperature of 180 degrees exhibiting superior performance at higher pressures. This outcome aligns with the expectations based on previous extensive laboratory experience. Notably, a corrective factor can be introduced to account for the volume of participating fibers in the polymer, offering analytical insights into the actual behavior of the final product.

The trend of tensile mechanical properties was supported by morphological observations of the surface and damage suffered by the panels studied. In agreement with this analysis, it is possible to state that the performances are certainly influenced by the level of compaction obtained for the structures but can be compromised by thermomechanical degradation phenomena to which biobased matrices and natural fibers are typically subject if non-optimal process conditions are adopted.

It is essential to highlight that this corrective factor is specific to the experiences presented in this study and is not applicable to all natural fibers, and in wider term to all fibers including the synthetic ones. Its applicability is confined to the hot press manufacturing technique and is limited to bi-directional woven fibers with 14 plies of fiber, excluding consideration for other manufacturing methods. For a comprehensive understanding, further investigations encompassing different number of plies, fiber types, manufacturing techniques, and validation against Finite Element Method (FEM) models are imperative to establish precise correlation factors for the impact of the manufacturing procedures on composite polymers.

CRediT authorship contribution statement

P. Russo: Writing – review & editing, Visualization, Validation, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **J. Passaro:** Visualization, Validation, Methodology, Investigation, Formal analysis. **A. Dib:** Writing – review & editing, Writing – original draft, Validation, Investigation, Formal analysis. **F. Fabbrocino:** Visualization, Supervision. **N. Fantuzzi:** Writing – review & editing, Visualization, Supervision, Software, Resources, Project administration, Methodology, Investigation, Funding acquisition, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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